

6.2 RECYCLE, RECOVERY, AND CONTROL TECHNOLOGIES ASSESSMENT

While pollution prevention is the preferred method of waste management, the waste management hierarchy recognizes that pollution prevention is not always feasible. Companies often supplement their pollution prevention efforts with additional waste management techniques to further reduce emissions. These techniques, presented in order of preference, include recycling, treatment, and disposal. This section presents waste management techniques typically used by the PWB industry in the MHC process to minimize waste, recycle or recover valuable process resources, and to control emissions to water and air.

6.2.1 Recycle and Resource Recovery Opportunities

PWB manufacturers have begun to reevaluate the merits of recycle and recovery technologies because of more stringent effluent pretreatment regulations. Recycling is the in-process recovery of process material effluent, either on-site or off-site, which would otherwise become a solid waste, air emission, or a wastewater stream. Metals recycling and recovery processes have become more economical to operate due to the increased cost of managing sludge containing heavy metals under stricter regulatory requirements. Technologies that recycle water from waste streams concentrate the final effluent making subsequent treatment more efficient, thus reducing the volume of waste generated along with overall water and sewer costs. As a result, these technologies are being used more frequently by industry to recycle or recover valuable process resources while also minimizing the volume of waste that is sent to disposal. This trend was supported by the respondents of the *Printed Wiring Board Pollution Prevention and Control: Analysis of Survey Results* (EPA, 1995c), 76 percent of whom reported using some type of recycle or resource recovery technology.

Recycle and resource recovery technologies include those that recover materials from waste streams before disposal or recycle waste streams for reuse in another process. Opportunities for both types of technologies exist within the MHC process. Rinse water can be recycled and reused in further rinsing operations while copper can be recovered from waste streams before disposal and sold to a metals reclaimer. These recycle and recovery technologies may be either in-line (dedicated and built into the process flow of a specific process line) or at-line (employed at the line as desired as well as other places in the plant) technologies depending on what is required (Brooman, 1996). Each individual waste stream that cannot be prevented should be evaluated to determine its potential for effective recycle or resource recovery.

The decision on whether to purchase a recycle or resource recovery process should be based on several factors. Economic factors such as process operating costs and effluent disposal costs for the current system must be compared with those estimated for the new technology. The initial capital investment of the new technology along with any potential cost savings and the length of the payback period must also be considered. Other factors such as the characteristics of the waste stream(s) considered for treatment, the ability of the process to accept reused or recycled materials, and the effects of the recycle or recovery technology on the overall waste treatment process should also be considered.

The entire PWB manufacturing process must be considered when assessing the economic feasibility of a recycle or resource recovery process. An individual recovery process can recover

copper from a single stream originating from the MHC process, or it may recover the metal from streams that originate from other processes as well. Only by considering the new technology's impact on the entire process, can an accurate and informed decision be made. While this section focuses on technologies that could be used to recycle or recover resources from the waste streams that are generated from the MHC process, many of these technologies are applicable to other PWB process lines. Workplace practices that can lead to the recycle or reuse of resources (e.g., manually recovering copper from panel racks, water recycle using cascade water rinse systems) are discussed in Section 6.1.

Reverse Osmosis

Reverse osmosis is a recovery process used by the PWB industry to regenerate rinse waters and to reclaim process bath drag-out for return to the process (EPA, 1990). It relies on a semi-permeable membrane to separate the water from metal impurities allowing bath solutions to be reused. It can be used as a recycling or recovery technology to reclaim or regenerate a specific solution, or it can be part of an overall waste treatment process to concentrate metals and impurities before final treatment.

The reverse osmosis process uses a semi-permeable membrane which permits only certain components to pass through it and a driving force to separate these components at a useful rate. The membrane is usually made of a polymer compound (e.g., nylon) with hole sizes ranging from 0.0004 to 0.06 microns in diameter. High pressure pumping of the waste stream, at pressures typically ranging from 300 to 1,500 pounds per square inch (psi) force the solution through the membrane (Capsule Environmental Engineering, Inc., 1993). The membrane allows the water to pass while inhibiting the metal ions, collecting them on the membrane surface. The concentrated metal ions are allowed to flow out of the system where they are reused as bath make-up solution or are sent to treatment. The relatively pure water can be recycled as rinse water or directly sewerred.

The reverse osmosis process has some limitations. The types of waste streams suitable for processing are limited to the ability of the plastic membranes to withstand the destructive nature of the given waste stream. The membranes are sensitive to solutions with extreme pH values, either low or high, which can degrade the polymer membranes. Pure organic streams are likewise not treatable. Waste streams with suspended solids should be filtered prior to separation to keep the solids from fouling the membrane, thus reducing the efficiency of the process. Process membranes may also have a limited life due to the long-term pressure of the solution on the membrane (Coombs, 1993). Data regarding the usage of reverse osmosis technology by industry was not collected by the Pollution Prevention Survey.

Ion Exchange

Ion exchange is a process used by the PWB industry mainly to recover metal ions, such as copper or palladium, from rinse waters and other solutions. This process uses an exchange resin to remove the metal from solution and concentrate it on the surface of the resin. It is particularly suited to treating dilute solutions, because it removes the metal species from the solution instead of removing the solution from the metal. As a result, the relative economics of the process improve as the concentration of the feed solution decreases. Aside from recovering copper, ion

exchange can also be used for treating wastewater, deionizing feed water, and recovering chemical solutions.

Ion exchange relies on special resins, either cationic or anionic, to remove the desired chemical species from solution. Cation exchange resins are used to remove positively charged ions such as copper. When a feed stream containing copper is passed through a bed of cation exchange resin, the resin removes the copper ions from the stream, replacing them with hydrogen ions from the resin. For example, a feed stream containing copper sulfate (CuSO_4) is passed through the ion exchange resin where the copper ions are removed and replaced by hydrogen ions to form sulfuric acid (H_2SO_4). The remaining water effluent is either further processed using an anion exchange resin and then recirculated into the rinse water system, or pH neutralized and then directly sewered. Ion exchange continues until the exchange resin becomes saturated with metal ions and must be regenerated.

Special chelating resins have been designed to capture specific metal ions that are in the presence of chelating agents, such as metal ions in electroless plating baths. These resins are effective in breaking down the chemical complexes formed by chelators that keep metal ions dissolved in solution, allowing them to be captured by the resin. They ignore hard water ions, such as calcium and magnesium that would otherwise be captured, creating a more pure concentrate. Chelating resins require that the feed stream be pH adjusted to reduce acidity and filtered to remove suspended solids that will foul the exchange bed (Coombs, 1993).

Regeneration of the cation or chelating exchange resin is accomplished using a moderately concentrated (e.g., ten percent) solution of a strong acid, such as sulfuric acid. Regeneration reverses the ion exchange process by stripping the metal ions from the exchange resin and replacing them with hydrogen ions from the acid. The concentration of metal ions in the remaining regenerant depends on the concentration of the acid used, but typically ranges from 10 to 40 g/L or more (Coombs, 1993).

Ionic exchange can be combined with electrowinning (electrolytic recovery) to recover metal from solutions that would not be cost-effective to recover using either technology alone. It can be used to concentrate a dilute solution of metal ions for electrolytic recovery that would otherwise be uneconomical to recover. For example, a dilute copper chloride solution can be treated by an ion exchange unit which is regenerated using sulfuric acid, producing a concentrated copper sulfate solution. The electrowinning unit can then be used to recover the copper from the solution while regenerating the acid, which could then be used for the next regeneration cycle.

A benefit of ion exchange is the ability to control the type of metallic salt that will be formed by selecting the type of acid used to regenerate the resin. In the previous example, the copper chloride was converted to copper sulfate while being concentrated by the ion exchange system. This is particularly useful when electrowinning is used, since it cannot process solutions containing the chlorine ion without generating toxic chlorine gas.

Twenty-six percent of the respondents to the Pollution Prevention Survey reported using an ion exchange process as a water recycle/chemical recovery technology. The average capital

cost of a unit, which is related to its capacity, reported by the respondents was \$47,500 with a low of \$5,000 and a high of \$100,000.

Electrolytic Recovery

Electrolytic recovery, also known as electrowinning, is a common metal recovery technology employed by the PWB industry. Operated either in continuous or batch mode, electrowinning can be applied to various process fluids including spent microetch, drag-out rinse water, and ion exchange regenerant. An advantage of electrowinning, which uses an electrolytic cell to recover dissolved copper ions from solution, is its ability to recover only the metal from solution without recovering the other impurities that are present. The recovered copper can then be sold as scrap or reused in the process.

Process waste solutions containing chlorine ions in any form should not be processed using electrolytic recovery methods since the electrolysis of these solutions could generate chlorine gas. Solutions containing copper chloride salts should first be converted using ion exchange methods to a non-chloride copper salt (e.g., copper sulfate) solutions before undergoing electrowinning to recover the copper content (Coombs, 1993).

Electrowinning is most efficient with concentrated solutions. Dilute solutions with less than 100 mg/L of copper become uneconomical to treat due to the high power consumption relative to the amount of copper recovered (Coombs, 1993). Waste streams that are to be treated should be segregated to prevent dilution and to prevent the introduction of other metal impurities. Already diluted solutions can be concentrated first using ion exchange or evaporation techniques to improve the efficiency and cost-effectiveness of metal recovery.

The electrolytic cell is comprised of a set of electrodes, both cathodes and anodes, placed in the copper laden solution. An electric current, or voltage, is applied across the electrodes and through the solution. The positively charged metal ions are drawn to the negatively charged cathode where they deposit onto the surface. The solution is kept thoroughly mixed using air agitation, or other proprietary techniques, which allow the process to use higher current densities (the amount of current per surface area of cathode) that speed deposition time and improve efficiency. As copper recovery continues, the concentration of copper ions in solution becomes depleted, requiring the current density to be reduced to maintain efficiency. When the concentration of copper becomes too low for its removal to be economically feasible, the process is discontinued and the remaining solution is sent to final treatment.

The layers of recovered copper can be sold as scrap to a metals reclaimer. Copper removal efficiencies of 90 to 95 percent have been achieved using electrolytic methods (EPA, 1990). The remaining effluent will still contain small amounts of copper and will be acidic in nature (i.e., low pH). Adjusting the pH may not be sufficient for the effluent to meet the standards of some POTW authorities; therefore, further treatment may be required.

Eighteen percent of the Pollution Prevention Survey respondents reported using electrowinning as a resource recovery technology with nearly all (89 percent) being satisfied. The median cost of a unit reported by the respondents was \$15,000; however, electrowinning capital costs are dependant on the capacity of the unit.

6.2.2 Control Technologies

If the release of a hazardous material cannot be prevented or recycled, it may be possible to treat or reduce the impact of the release using a control technology. Control technologies are engineering methods that minimize the toxicity and volume of released pollutants. Most of these methods involve altering either the physical or chemical characteristics of a waste stream to isolate, destroy, or alter the concentration of target chemicals. While this section focuses on technologies that are used to control on-site releases from the MHC process, many of these technologies are also applicable to other PWB process lines.

Control technologies are typically used to treat on-site releases to both water and air from the operation of the MHC process. Wastewater containing concentrations of heavy metal ions, along with chelators and complexing agents, are of particular concern. Water effluent standards require the removal of most heavy metals and toxic organics from the plant effluent before it can be disposed to the sewer. On-site releases to air of concern include formaldehyde vapors, as well as acid and solvent fumes. The desire to eliminate both formaldehyde and chelating agents has led to the development of alternative MHC processes. This section identifies the control technologies used by PWB manufacturers to treat or control wastewater and air emissions released by the operation of the MHC process.

Wastewater Treatment

Chemical Precipitation. In the PWB industry, the majority of facilities surveyed (61 percent) reported using a conventional chemical precipitation system to accomplish the removal of heavy metal ions from wastewater. Chemical precipitation is a process for treating wastewater that depends on the water solubility of the various compounds formed during treatment. Heavy metal cations that are present in the wastewater are reacted with certain treatment chemicals to form metal hydroxides, sulfides, or carbonates that all have relatively low water solubilities. The resulting heavy metal compounds are then precipitated from the solution as an insoluble sludge that is subsequently recycled to reclaim the metals content or sent to disposal. The chemical precipitation process can be operated as a batch process, but is typically operated in a continuous process to treat wastewater.

In the chemical precipitation treatment of wastewater from PWB manufacturing, the removal of heavy metals may be carried out by a unit sequence of rapid mix precipitation, flocculation, and clarification. The process begins with the dispersion of treatment chemicals into the wastewater input stream under rapid mixing conditions. The initial mixing unit is designed to create a high intensity of turbulence in the reactor vessel, promoting encounters between the metal ions and the treatment chemical species, which then react to form metal compounds that are insoluble in water. The type of chemical compounds formed depends on the treatment chemical employed; this is discussed in detail later in this section. These insoluble compounds form a fine precipitate at low pH levels that remains suspended in the wastewater.

The wastewater then enters the flocculation tank. The purpose of the flocculation step is to transform smaller precipitation particles into large particles that are heavy enough to be removed from the water by gravity settling in the clarification step. This particle growth is accomplished in a flocculation tank using slow mixing to promote the interparticle collisions of

precipitate particles suspended in the wastewater. The degree of flocculation is enhanced through the use of flocculating chemicals such as cationic or anionic polymers. These chemicals promote interparticle adhesion by adding charged particles to the wastewater that attach themselves to the precipitate, thereby increasing the growth rate of the precipitate particles.

Clarification is the final stage of the wastewater treatment process. The wastewater effluent from the flocculation stage is fed into a clarification tank where the water is allowed to collect undisturbed. The precipitate then settles out of the water by gravity, forming a blanket of sludge at the bottom of the clarification tank. A portion of the sludge, typically 10 to 25 percent, is often recirculated to the head of the flocculation step to reduce chemical requirements, as well as to enhance the rate of precipitation (Frailey, 1996). The sludge particles provide additional precipitation nuclei that increase the probability of particle collisions, resulting in a more dense sludge deposit. When a dense layer of sludge has been formed, the sludge is removed from the tank and is either dewatered or sent for recycle or disposal. The precipitate-free water is then either recycled or sewered.

Other process steps are sometimes employed in the case of unusually strict effluent guidelines. Filtration, reverse osmosis, ion exchange, or additional precipitation steps are sometimes employed to further reduce the concentration of chemical contaminants present in the wastewater effluent.

The heavy metal sludge generated by the wastewater treatment process is often concentrated, or dewatered, before being sent to recycle or disposal. Sludge can be dewatered in several methods including sludge thickening, press filtration, and sludge drying. Through the removal of water, sludge volume can be minimized, thus reducing the cost of disposal.

Treatment of Non-Chelated Wastewater. The absence of complexing chemicals (e.g., ammonia) or chelating agents (e.g., EDTA) in the wastewater stream simplifies the removal of heavy metal ions by precipitation. Heavy metal removal from such waste streams is accomplished through simple pH adjustment using hydroxide precipitation. Caustic soda (NaOH) is typically used while other treatment chemicals include calcium hydroxide and magnesium hydroxide. The heavy metal ions react with the caustic soda to form insoluble metal hydroxide compounds that precipitate out of solution at a high pH level. After the precipitate is removed by gravity settling, the effluent is pH adjusted to a pH of seven to nine and then sewered. The treatment can be performed in a chemical precipitation process similar to the one described above, resulting in a sludge contaminated with metals that is then sent to recycling or disposal.

Treatment of Wastewater Containing Chelated Metals. The presence of complexing chemicals or chelators require a more vigorous effort to achieve a sufficient level of heavy metal removal. Chelators are chemical compounds that inhibit precipitation by forming chemical complexes with the metals, allowing them to remain in solution beyond their normal solubility limits. These chemicals are found in spent MHC plating baths, in cleaners, and in the water effluent from the rinse tanks following these baths. Treatment chemicals enhance the removal of chelated metals from water by breaking the chelant-to-metal bond, destroying the soluble complex. The freed metal ions then react to form insoluble metal compounds, such as metal hydroxides, that precipitate out of solution. Several different chemicals are currently being used

to effectively treat chelator-contaminated wastewater resulting from the manufacture of PWBs. Some common chemicals used in the treatment of wastewater produced by the MHC process are briefly described in Table 6.6. For a more information regarding individual treatment chemicals and their applicability to treating specific wastes, consult the supplier of the treatment chemical.

Table 6.6 Treatment Chemicals Used to Remove Heavy Metals From Chelated Wastewater

Chemical	Description
Ferrous Sulfate	Inexpensive treatment that requires iron concentrations in excess of 8:1 to form an insoluble metal hydroxide precipitate (Coombs, 1993). Ferrous sulfate is first used as a reducing agent to breakdown the complexed copper structures under acidic conditions before forming the metal hydroxide during subsequent pH neutralization. Drawbacks include the large volumes of sludge generated and the presence of iron which reduces the value of sludge to a reclaimer.
DTC (Dimethyl-dithiocarbamate)	Moderately expensive chemical that acts as a complexing agent, exerting a stronger reaction to the metal ion than the chelating agent, effectively forming an insoluble heavy metal complex. The sludge produced is light in density and difficult to gravity separate (Guess, 1992; Frailey, 1996).
Sodium Sulfide	Forms heavy metal sulfides with extremely low solubilities that precipitate even in the presence of chelators. Produces large volume of sludge that is slimy and difficult to dewater (Guess, 1992).
Polyelectrolyte	Polymers that remove heavy metals effectively without contributing to the volume of sludge. Primary drawback is the high chemical cost (Frailey, 1996).
Sodium Borohydride	Strong reducing agent reduces heavy metal ions which then precipitate out of solution forming a compact, low volume sludge. Drawbacks include its high chemical cost and the evolution of potentially explosive hydrogen gas (Guess, 1992; Frailey, 1996).
Ferrous Dithionite	Reduces heavy metal ions under acidic conditions to form metallic particles that are recovered by gravity separation. Excess iron is regenerated instead of being precipitated producing a low volume sludge (Guess, 1992).

Effects of MHC Alternatives On Wastewater Treatment. The strong desire to remove both formaldehyde and complexing chemicals, such as chelators, from the MHC process has led the drive away from traditional electroless copper and toward the development of alternative MHC processes. These processes eliminate the use of chelating agents that inhibit the precipitation of heavy metal ions in wastewater. Also eliminated is the need for expensive treatment chemicals, which are designed to breakdown chelators and which can add to the quantity of sludge produced. The resulting treatment of the non-chelated waste stream produces less sludge at a lower chemical treatment cost than it would if chelators were present. A detailed description of the treatment for both chelated and non-chelated wastes is presented elsewhere in this chapter.

While MHC alternative processes may reduce or eliminate the presence of chelators in the wastewater, they do not create any additional treatment concerns that would require any

physical changes in the treatment process. The treatment of wastewater generated from the operation of a MHC alternative can be accomplished using the traditional chemical precipitation stages of rapid mix precipitation, flocculation, and clarification.

Alternative Treatment Processes. Although chemical precipitation is the most common process for treating wastewater by PWB manufacturers, other treatment processes exist as well. Survey respondents reported the use of both ion exchange (33 percent) and/or electrowinning (12 percent) to successfully treat wastewater generated from the manufacture of PWBs. These processes operate separately, or in combination, to efficiently remove heavy metal ions from chelated or non-chelated waste streams, typically yielding a highly concentrated sludge for disposal. These processes were discussed in Section 6.2.1.

Batch Treatment of Process Baths. Most spent process baths can be mixed with other wastewater and treated by the on-site wastewater treatment process using chemical precipitation. Chemical suppliers, however, recommend that some process baths be treated separately from the usual waste treatment process. The separate treatment of these baths is usually recommended due to the presence of strong chelating agents, high heavy metal concentrations, or other chemicals, such as additives or brighteners, that require additional treatment measures before they can be disposed of properly. Spent bath solution requiring special treatment measures can be processed immediately, but is typically collected and stored until enough has accumulated to warrant treatment. Batch treatment of the accumulated waste is then performed in a single tank or drum, following the specific treatment procedures provided by the chemical supplier for that bath.

Despite the supplier's recommendations, PWB facilities sometimes treat individual process baths using their typical wastewater treatment process. Spent bath solutions can be mixed slowly, in small quantities, with other wastewater before being treated, thus diluting the concentration of the chemical species requiring treatment. However, the introduction of concentrated wastes to the wastewater could result in increased treatment chemical consumption and more sludge produced than if batch treated separately. Also the introduction of a chemical species not typically found in the wastewater may adversely affect the treatment process or require more vigorous treatment chemicals or processes. Factors affecting the success of such treatment include the type of treatment chemicals used, the contaminant concentrations in the wastewater, and the overall robustness of the treatment process.

Air Pollution Control Technologies

Air pollution control technologies are often used by the PWB industry to cleanse air exhaust streams of harmful fumes and vapors. Exactly half (50 percent) of the PWB facilities surveyed have installed air scrubbers to control air emissions from various manufacturing processes, and almost a quarter of the facilities (23 percent) scrub air releases from the MHC process. The first step of any air control process is the effective containment of fugitive air emissions at their source of release. This is accomplished using fume hoods over the process areas from which the air release of concern is emanating. These hoods may be designed to continuously collect air emissions for treatment by one of the methods described below.

Gas Absorption. One method for removing pollutants from an exhaust stream is by gas absorption in a technique sometimes referred to as air scrubbing. Gas absorption is defined as the transfer of material from a gas to a contacting liquid, or solvent. The pollutant is chemically absorbed and dispersed into the solvent leaving the air free of the pollutant. The selection of an appropriate solvent should be based upon the liquid's solubility for the solute, and the cost of the liquid. Water is used for the absorption of water soluble gases while alkaline solutions are typically used for the absorption of acid gases. Air scrubbers are used by the PWB industry to treat wet process air emissions, such as formaldehyde and acid fumes, and emissions from other processes outside the MHC process.

Gas absorption is typically carried out in a packed gas absorption tower, or scrubber. The gas stream enters the bottom of the tower, and passes upward through a wetted bed of packing material before exiting the top. The absorbing liquid enters the top of the tower and flows downward through the packing before exiting at the bottom. Absorption of the air pollutants occurs during the period of contact between the gas and liquid. The gas is either physically or chemically absorbed and dispersed into the liquid. The liquid waste stream is then sent to water treatment before being discharged to the sewer. Although the most common method for gas absorption is the packed tower, other methods exist such as plate towers, sparged towers, spray chambers, or venturi scrubbers (Cooper, 1990).

Gas Adsorption. The removal of low concentration organic gases and vapors from an exhaust stream can be achieved by the process of gas adsorption. Adsorption is the process in which gas molecules are retained on the interface surfaces of a solid adsorbent by either physical or chemical forces. Activated carbon is the most common adsorbent but zeolites such as alumina and silica are also used. Adsorption is used primarily to remove volatile organic compounds from air, but is also used in other applications such as odor control and drying process gas streams (Cooper, 1990). In the MHC process it can be used to recover volatile organic compounds, such as formaldehyde.

Gas adsorption occurs when the vapor-laden air is collected and then passed through a bed of activated carbon, or another adsorbent material. The gas molecules are adsorbed onto the surface of the carbon, while the clean vapor-free air is exhausted from the system. The adsorbent material eventually becomes saturated with organic material and must be replaced or regenerated. Adsorbent canisters, which are replaced on a regular basis, are typically used to treat small gas flow streams. Larger flows of organic pollutants require packed beds of adsorbent material, which must be regenerated when the adsorbent becomes saturated (Cooper, 1990).

Regeneration of the adsorbent is typically accomplished by a steam stripping process. The adsorbent is contacted with low pressure steam which desorbs the adsorbed gas molecules from the surface of the packed bed. Following condensation of the steam, the organic material is recovered from the water by either decanting or distillation (Campbell, 1990).

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